CLASSIFICATION TESTS FOR ASSIGNMENT TO HAZARD CLASS/DIVISION 1.6: SNPE TWO YEARS EXPERIENCE

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ABSTRACT:

The UN Test series 7 can be considered as the most complete, and severe, list of requirements for insensitive munitions. So, it has been extensively used at SNPE, as a screening method for the new high explosives formulations studies. This two years experience provided us valuable information to analyse the pertinence of the substances tests [7(a) to 7(f)], from different points of view:

- at first, the equivalence of distinct ways permitted for a same test has been looked (high explosive donor for Gap Test, Susan Test/Friability Test comparison)
- secondly, some experimental procedures may be discussed, at the light of results and observations during tests. (External Fire Test, Slow Cook-off Test, Bullet Impact Test)
- finally, the knowledge of EIDS behavior in large scale models or in warheads, allows to confirm the substances tests interest, while also indicating some of their limitations.

This three points are discussed in the paper, with the support of results and observations provided by assessments of EIDS candidates, as well as expected advantages with C/D 1.6.

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1) INTRODUCTION

The new hazard class/division (C/D) 1.6, for articles, explosives, extremely insensitive, has been first created by the transportation community, under the impulse of the United Nations Comitee of Experts on the Transport of Dangerous Goods.

This Comitee approved a new test protocol (Test Series 7), which is described in ref [1]. The tests and pass/fail criteria required by the test series 7 are briefly summarized in appendix 1:

- Tests 7a) to 7f) for substances, to "qualify" a new material as an "Extremely Insensitive Detonating Substance" (EIDS), or "Matière Détonante Extremement Peu Sensible" (MDEPS) in French.
- Tests 7g) to 7k), for articles, to assign a classification in C/D 1.6 to a new ammunition, containing only EIDS as high explosives.

Since that time, the C/D 1.6 has been adopted in other regulations or classification procedures :

- by the US DOT, in its new document 49 CFR, vol. 55, n° 246
- by the US DOD, in DOD 6055-9-STD, change n° 3, which adopted the EIDS and C/D 1.6 appellations, but not yet the UN tests.
- by the NATO AC/258, in a draft AASTP3 (Allied Ammunition Storage and Transport Publication n° 3)

Then this new C/D is progressively on the way to become a standard for hazard classification of insensitive munitions, concerning both storage and transportation.

So SNPE has been interested, for more than two years now, in using this protocol to assess its new insensitive high explosives.

This experience has been used, and analysed, to improve the knowledge about the new EIDS materials, concerning mainly the following topics:

- the significance of the tests and criteria for EIDS, which have already been widely described in former presentations [2,3].
- the differences between the allowed alternatives methods,
- the consequences and limitations of the pass/fail criteria,
- the expected advantages for C/D 1.6 ammunition.

2) SUBSTANCES TESTS:

2.1. EIDS Cap Test - 7a) :

This test is used to assess the sensitivity of an EIDS candidate to a standardized detonator, the sample being an unconfined cylinder (80mm diameter, 160mm length). Results obtained with this procedure are presented in table n° 1.

In this table, and in all others coming, (-) means the acceptance criterion is met, while (+) means it is not.

Some details about the test substances in this tables are also given in appendix 2.

Test substance	Result	Data reference	Remarks : unconfined critical diameter (mm)
TNT	(-)	SNPE	40
Compo. B	(+)	US [4]	4.3
PBX 9502	(-)	US [4]	8-10
CPX 305	(-)	UK/DRA [5]	42-47
AFX 644	(-)	US/EGLIN [6]	
ORA 86	(-)	SNPE	4
B2214	(-)	SNPE	65
B2211	(-)	SNPE	65
B3017	(-)	SNPE	10-15
B3021	(+)	SNPE	< 10

Table no 1: Cap Test results ((-) means no detonation)

In a first approximation, this test selects the materials mainly according to their critical diameter:

- a critical diameter larger than 10mm will surely lead to pass the test.
- but some negative results may also be obtained with cast PBX having smaller critical diameter than 10mm.

2.2. EIDS Gap Test - 7b) :

This test is used to assess the shock sensitivity of an EIDS candidate (73mm diameter, 280mm length, 11mm steel confinement) which is subjected to a shock pulse, delivered by a donor charge through a PMMA barrier.

The UN protocol allows to use:

- either a Pentolite 50/50 donor,
- or a RDX/Wax 95/5 donor.

Pressure measurements have been done at SNPE with the RDX/Wax donor. They are compared with NSWC data on the figure in appendix 3, which shows that the two donors give roughly the same peak pressure, especially near the 70mm criterion, and then can be considered as equivalent for comparisons between Gap Test data.

Results obtained at SNPE are gathered in table no 2, as well as some other data.

Test substances	Result	Critical PMMA thickness (mm)	Critical equivalent pressure(GPa)	Data reference
TNT TNT-AI 85/15 HBX3 Compo. B Octol 75/25 ORA 86 B2214 B2211 B3017	(+) (+) (+) (+) (+) (+) (-)	110 95 110 155 195 90 35 50	1.8 2.7 2.0 0.7 0.2 3.5 9.5 8.0	SNPE " " " " " "
B3017	(-)	35	10.0	n
B3021	(-)	< 70	> 4.0	
PBX 9502	(-)	52	7.0	US/NSWC [4]
CPX 305	(+)	76		UK/DRA [5]
AFX 644	(-)	52		US/EGLIN AFB [6]

Table n° 2: Gap Test results ((-) means no detonation with 70mm PMMA)

According to the calibration curves in appendix 3, the candidates meeting the pass/fail criterion at 70mm PMMA, have a shock sensitivity higher than about 4.0 GPa, measured in the 73mm diameter. We will see later in the paper how this selection is pertinent when talking about sympathetic detonation.

2.3. EIDS Impact sensitivity - 7c) and 7d):

The assessment of impact sensitivity is the only point of the protocol where two alternative methods are offered:

- the US proposal has been to perform the Susan Test 7c)i) and a Bullet Impact test 7d)i),
- the French proposal has been to perform only the Friability test 7c)ii) and 7d)ii).

At this time both of these procedures are used respectively in US and in France. Then some additional work have been undertaken in US (DDESB and NSWC) and in France (STPE and SNPE), in order:

- to have a recognition of the French alternative method in US,
- to study the possibility of reducing the number of test for assessing the impact sensitivity.

So answers have been searched for the two questions asked by this alternativity:

- Is the Friability test equivalent to Bullet Impact Test?
- Is the Friability test equivalent to Susan Test?

To the first question, the answer is definitely yes. This can be easily demonstrated by all the works made by SNPE concerning the DDT hazard due to bullet impact on confined warhead [7,8,9]. The table in appendix 4 summarizes these works, by comparing for sixteen high explosives:

- their behavior at 0.5 caliber bullet impact in a 20mm steel confinement generic unit, with inside diameter 125 mm, and length 90 mm.
- their Friability level.

It is clear from these results that a detonation hazard to 0.5 bullet impact may not be expected any more with cast PBX having a Friability level lower than 15 MPa/ms.

Concerning the second question, results obtained by both Friability test and Susan Test are gathered in table no 3.

Test	Friability test			Susan Test		
substances	MPa/ms at 150 m/s	Result	Data ref.	ΔP or TNT equiv. at 333m/s	Result	Data ref.
TNT TNT-Al 85/15 HBX3 Compo. B Octol 75/25 ORA 86 B3103 B3003 B2214 B2211 B3017 B3021	7.8 6.5 3.0 51 > 33 8.0 3.9 > 26.5 0.2 4.5 1.3 7.5	(-) (-) (+) (+) (-) (-) (-) (-) (-)	SNPE	17.7KPa 190g (80/20) / 32KPa > 250g 25KPa 46KPa 43KPa 4.9KPa /	(-) (+) (+) (+) (-) (+) (-) /	UK/DRA[1] DOBRATZ[11] US/NSWC[4] DOBRATZ[11] US/NSWC[4] US/NSWC[4] US/NSWC[4] US/NSWC[4] UK/DRA[11] /
PBX9502 CPX 305 AFX 644 AFX 930 AFX 931	/ 0.2 0.3 /	/ (-) (-) /	/ " ' /	6.7KPa 11.1KPa 17.1KPa 12.0KPa 25.0KPa	(-) (-) (-) (-)	US/NSWC[4] UK/DRA[6] US/EGLIN[7] US/NSWC US/NSWC

Table no 3: Results to Friability test and Susan Test ((-) means less than 15 MPa/ms at Friability test, or less than 27KPa or 45 g TNT at Susan Test).

These comparisons show some discrepancy between the two methods in only two cases: TNT-Al and B 3103. In each of these two cases the substance passes the Friability criterion and fails the Susan Test criterion. Our explanation to these differences is that in the Friability test, the substance is first impacted, being only damaged but not burning, and is secondly burned in a clossed vessel.

On the contrary, the Susan Test may be considered as well as a shock sensitivity test and as an impact test, since the substance is reacting at impact.

That's why we suggest that, in the mind of the Test Series 7 for substances, those results would no be considered separately but rather all together, especially by taking into account the Gap Test result.

By doing so it can be demonstrated that both the two alternative methods lead to the same global verdict, as shown by the next table.

Test substances	Friability result	Susan result	Gap Test result	Friability + Gap Test global result	Susan + Gap Test global result
TNT Compo. B TNT-AI 85/15 AFX 644 PBX 9502 CPX 305 HX 76 B2214 ORA 86 B3103 B3003	(-) (+) (-) (-) (-) (-) (-) (-)	(-) (+) (+) (-) (-) (-) (-) (+) (+)	(+) (+) (+) (-) (-) (+) (+) (-) (+) (+)	(+) (+) (+) (-) (-) (+) (+) (-) (+)	(+) (+) (+) (-) (-) (+) (+) (-) (+) (+)

Table no 4: Global comparison between Friability - Susan - Gap Test

The reason for that is: since the Susan Test may be considered also as a shock sensitivity test, then the Gap Test will filter more severely the candidates in that way, as shown by the figure in appendix 5 where:

- 3/12 substances fail to both Susan and Gap Tests
- 5/12 substances pass both Susan and Gap Tests
- 4/12 substances pass the Susan Test but fail at Gap Test.
- No substance passes the Gap Test while failing the Susan Test.

2.4. EIDS External Fire Test - 7e)

This test has the objective of indicating the reaction level at fast cookoff of a substance contained in a 4mm thickness steel pipe (40mm ID, 200mm length) ended by two steel caps.

The test 7e) consists in putting five of these pipes in a fuel fire.

In France the test is performed differently at this time:

- either with only one pipe, for the substances having a small critical diameter
 - or with a 3 liters confined generic unit, for substances with larger critical diameter. The sample has then a 123mm diameter.

Some results obtained at SNPE and GERBAM* by these two methods are presented in the next table.

Test	40mm pir	Эе	3 liters generic unit		
substances	Reaction level	Result	Reaction level	Result	
TNT TNT-Al85/15 Compo. B Octol HBX3 ORA 86 B2214 B2211 B3017	Detonation Pressure burst Detonation Detonation Pressure burst Pressure burst / / /	(+) (-) (+) (+) (-)	Violent Reaction Pressure burst Partial deto. Partial deto. Pressure burst Pressure burst Pressure burst Pressure burst Pressure burst Pressure burst	(+) (-) (+) (+) (-) (-) (-) (-)	

Tableau n° 5: External fire results ((-) means no detonation and no violent reaction)

Similar responses are obtained in both those two containers in fuel fire. On another hand, we observed that when performing the test with five pipes, as recommended by the UN procedure, only one pipe reacts, the others being expelled out of the fire. Then now, only one pipe is tested.

^{*} Groupe d'Etudes et de Recherhes en Balistique des Armes et Munitions - DCN Lorient

2.5. EIDS Slow Cookoff Test - 7f):

This test is used to determine the reaction of an EIDS candidate when heated in an oven at 6 F/hr (3,3°C/hr), in the same pipe as for test 7e).

The next table presents some results obtained at SNPE with the UN procedure, and at GERBAM in the 3 liters generic unit.

Test	40mm pipe		3 liters generic unit		
substances	Reaction level	Result	Reaction level	Result	
TNT Compo. B TNT-Al85/15 ORA 86 B2214 B2211 B3017 B3021	Violent reaction / / Pressure burst Pressure burst Pressure burst Violent reaction Detonation	(+) (-) (-) (-) (+) (+)	Violent Reaction Detonation Pressure burst Pressure burst Pressure burst Pressure burst Pressure burst Pressure burst	(+) (+) (-) (-) (-) (-)	

Table 6: Slow Cookoff results ((-) means no detonation and no violent reaction)

These results show some discrepancy between the two methods, which can be explained by the interpretation of the pipe fragmentation given in the UN protocol. A reaction is considered as violent as soon as the pipe fragments in more than three pieces, without making any difference between:

- a complete fragmentation of the pipe, due to a violent reaction,
- a fragmentation of only one part of the pipe, generally near one of the two caps, which may be the result of a pressure burst.

3 - ARTICLES TESTS:

Having much less experience with articles tests than with substances tests we can only, in that part of the paper, analyse the validity of pass/fail criteria, and their consequences, for predicting the behavior of a munition. In fact, what we consider as important is the interest of the substances tests in preventing the use, in C/D 1.6 articles, of high explosives with intrinsical properties not sufficient to guarantee an intrinsical safety of the munition.

3.1. External Fire:

The expected behavior for passing the criterion is a reaction no more severe than a burning, which, as shown by the hazard analysis protocol we have established for fuel fire response (see appendix 6), can be reached by several ways:

- (1) by a temperature deconfinement of the case,
- 2 by a pressure deconfinement of the case before ignition, due to pyrolysis gases,
- 3 or, in case of ignition, by having a high explosive which will burn slowly enough to only mildly break the case.

The EIDS External Fire Test 7e) will provide information for points ② and ③, but only in a medium confinement system (4mm steel).

Then it can be considered as a good screening test for high explosives candidates for C/D 1.6 articles with medium or high confinements, since it reproduces well the physical behavior allowing a smooth response of such an ammunition. But on the contrary, this filter may be severe for low confinement munitions, and for temperature degradable structures.

3.2. Slow Cookoff:

The mechanisms playing a role for the munition response to such a stimulus are more complex than for the fuel fire.

The works performed at SNPE for some years allowed us to establish a draft of hazard analysis protocol, based on the fuel fire protocol (see appendix 7).

A reaction no more severe than a burning may also be expected by several ways:

- 1 by a temperature deconfinement of the case, which is very unlikely by heating at only 6F/hr
- 2 by a pressure deconfinement of the case before ignition, due to pyrolysis gases
- ③ or, in case of ignition, by having a high explosive which will burn slowly enough to only mildly break the case, but this after a "cooking" of more than 25 hours.

The EIDS Slow Cookoff Test 7f) will provide informations for points ② and ③, but also only in a medium confinement system (4mm steel).

Then once again this test can be considered as a good screening test for high explosives candidates for C/D 1.6 articles, with the same observations than for fuel fire, regarding the confinement.

3.3. Bullet Impact:

According to the protocol in appendix 8, and as shown by all the works performed at SNPE for more than ten years about this topic [9, 12], two of the EIDS tests are representative of the most important parameters regarding to the hazard of detonation or violent reaction to bullet impact, which is dependent on:

- 1) the ignitability of the high explosive, which isn't taken into account by any of the EIDS tests. But the absence of reaction is not a requirement for C/D 1.6.
- 2) the mechanical behavior to high rate sollicitation and the quickness of the damaged material, which are both covered by the Friability Test. Both of these properties are very important in the process of transition from burning to deflagration, and to detonation.
- 3) the ability of the high explosive to burn layer by layer, even at pressures as high as some Kbar. We generally are used to assess this behavior by burning in high pressure closed vessel [8]. But the EIDS External Fire test has also be found able to assess that property, since a mild reaction at this test is only possible with high explosives remaining compact during burning under confinement.

Then among the EIDS tests, the Friability and External Fire tests are together good filters to screen high explosives candidates for C/D 1.6 articles, with no violent reaction to bullet impact.

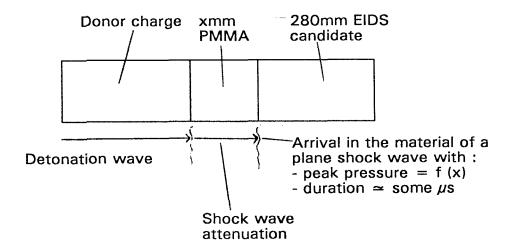
For example, in the case of a heavily confined warhead:

- 5 < Friability < 20 MPa/ms ---> good chance of pneumatic burst
- Friability < 5 MPa/ms ---> good chance of burning only

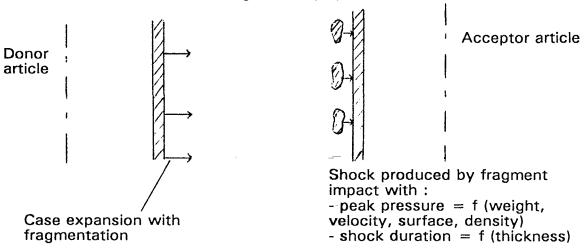
3.4. Detonation Propagation:

The Gap Test is the only EIDS test acting as a filter regarding to the hazard of detonation propagation.

But if we look at the description of that test, we can see that the EIDS candidate is only assessed regarding to its initiation in detonation by a plane shock wave generated through an inert material:



The situation is different when talking about sympathetic detonation:



The sympathetic detonation process is then not only dependent on the high explosive shock sensitivity, but also on the energy put into the fragments by the donor charge.

These figures only summarise the difficulty in simply correlating the Gap Test data to the sympathetic detonation process. But some results available now allow nevertheless to better precise the limitation of such an interpretation.

For example, in the case of articles with 12.5mm thickness steel structure, the figure in appendix 9 shows which kind of information can be expected from the EIDS Gap Test.

So, in such a configuration (1 donor/1 acceptor, 12.5mm steel confinement naturally fragmentated), an EIDS candidate which just passes the 70mm PMMA criterion could no sympathetically detonate in articles with only diameters less than 110mm.

But this interpretation must be taken with care since it doesn't take into account many important aspects of the problem:

- the stack effects due to higher confinement,
- the notions of run distance and critical diameter,
- the presence of a booster,
- shock waves reflections which can occur in a munition.

So, the EIDS Gap Test occurs as a filter not too severe with the 70mm PMMA criterion ($\simeq 4.0$ GPa), and as less severe as the confinement will be heavy and the article diameter will be large.

4 - CONCLUSIONS

The number of data obtained by performing the test series 7 from the UN protocol for C/D 1.6 has now reach a level sufficient to better know what means exactly that protocol:

- 1° The EIDS tests have been demonstrated to well exhibit the hazardous behaviors of known unsafe high explosives: mainly compo.B and Octol, and at a lower degree TNT, Tritonal and HBX3.
- 2° On another hand, regarding to the impact sensitivity assessment, the two alternative methods lead globally to the same verdict, when taken together with the Gap Test.
- 3° As a consequence we can now figure more precisely what looks like a true EIDS, with the key point being to pass the Gap Test criterion:
 - not sensitive to the standard detonator (Dc > 10mm)
 - no go at Gap Test behind 70mm PMMA (Pi > 4.0 GPa)
 - Friability less than 5 MPa/ms at 150m/s, or Susan result less than 10KPa at 333m/s and no more than burning at 0.5 caliber bullet impact.
 - no more than pneumatic burst at fast cookoff in confined pipe (then burning layer by layer under high pressure)
 - no more than pneumatic burst at slow cookoff in confined pipe (burning layer by layer under high pressure after "cooking").

SNPE can propose at this time two true EIDS:

- B 2211 for underwater and air blast applications,
- B 2214 for naturally fragmented warheads. Some others candidates are on the way to pass all the criteria, with higher performances.
- 4° Among the five EIDS tests, four have been found very pertinent as screening tests before performing the article tests:
 - the EIDS tests for fuel fire, slow cookoff and bullet impact are well fitting with hazard analysis protocols established by SNPE
 - the EIDS Gap Test has to be interpreted carefully, because of the limitations about its meaning as a function of the important parameters playing a role in the sympathetic detonation process.
 - the Cap Test has not been found relevant of any accidental situation.

As a conclusion:

- the use of EIDS is probably the best way of offering loadings with intrinsical properties which can guarantee an intrinsical safety of a munition. In some cases this guarantee could probably seem at a higher level than what could be required for the munition (munitions with very low confinement). But on another hand, those munitions would be severely damaged in a disaster, and then require more intrinsically safe substances.
- the four article tests are well in accordance with most of the other IM requirements, although some adaptations could be interesting in order to be fully similar (with STANAG for example). Even if those requirements are narrowly specified (0.5 caliber bullet at 850 m/s for example), the use of EIDS allows to expect good behaviors in a larger range of stimulus (for instance with tumbling bullets).

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Appendix 1: C/D 1.6 Test Series (from [3])

TEST	NAME OF TEST	COUNTRY OF	FAILURE
NUMBER		ORIGIN	CRITERIA
TESTS ON SU	BSTANCES		
7(a)	EIDS CAP TEST	Germany/US	Detonation of any sample
7 (b)	EIDS GAP TEST	US	Detonation at gap of 70 mm
7(c) (i)	SUSAN TEST	US	P>27kPa @ v=333 m/s
7 (c)(ii)	FRIABILITY TEST	France	dp/dt >15 MPa/ms for v=150 m/s
7(d) (i)	EIDS BULLET IMPACT TEST	US	Explosion/Detonation
7(d)(ii)	FRIABILITY TEST	France	dp/dt >15 MPa/ms for v=150 m/s
7(e)	EIDS EXTERNAL FIRE TEST	UN	Detonation, fragment throw > 15 m
7(f)	EIDS SLOW COOK-OFF TEST	us	Detonation, > 3 fragments
TESTS ON AR	TICLES		
7(g)	1.6 ARTICLE EXTERNAL FIRE TEST	l un	C/D 1.1, 1.2, or 1.3 response
7(h)	1.6 ARTICLE SLOW COOK-OFF TEST	US	Reaction > burning
70)	1.6 ARTICLE BULLET IMPACT TEST	US	Detonation
7(k)	1.6 ARTICLE STACK TEST	UN	Propagation
		·	•

Appendix 2: Details on SNPE test substances

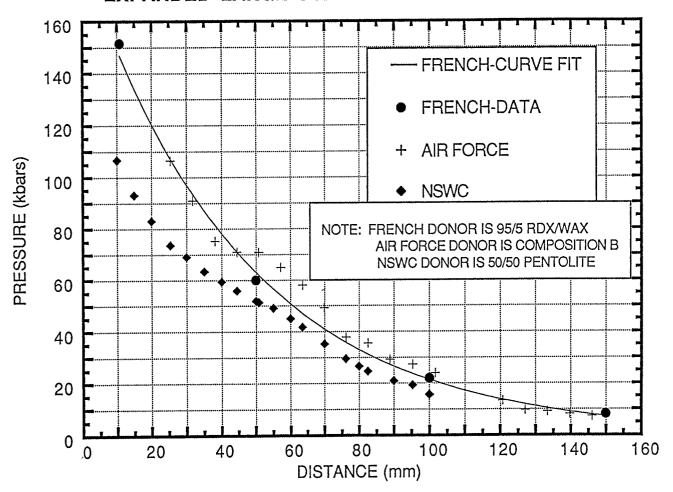
- Inert binder PBXs: - Energetic binder PBXs:

B3003 : HMX

ORA 86 : HMX B2214 : HMX - NTO B2211 : RDX - AP - AI B3103 : HMX - Al

B3017 : NTO B3021 : HMX - NTO

EXPANDED LARGE SCALE GAP TEST CALIBRATION



Appendix 4: Friability and Bullet Impact data

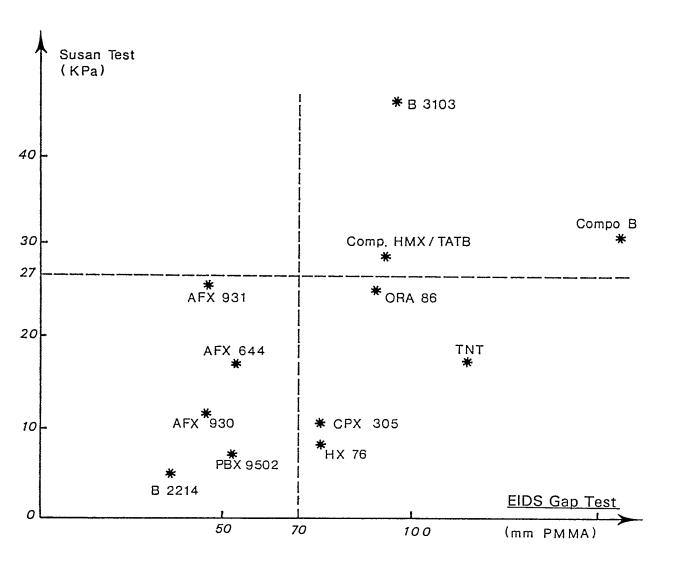
DEECDENOE	COMPOSITION	BULLI	ET IMP	ACT	(m/s)	FRIABILITY
REFERENCE		740	810	930	1140	Dmax (MPa/ms) at $V = 150 \text{ m/s}$
HEXOLITE 65/35	- TNT - RDX	D	D	Q	D	114
E1	- PU - HMX	Е	Е	E	D	51
E2	- PU - HMX	Е	Е	Е	D	42
E3 (B 3003)	- G - HMX	D	D	D	D	26,5 at V = 87 m/s
E4	- PU - HMX	A	Е	Е	D	24 –
E5	- PU - HMX	E	Е	Е	D	21,6
E6	- PB - RDX			DEF	DEF	17
E7	- PB - HMX		DEF	DEF	DEF	15,4
E8 (ORA 86A)	- PU - HMX	A	DEF	DEF	DEF	8,0
E9	- PB - HMX		DEF	DEF	DEF	6,7
E10	- PB - RDX - Alu	DEF	DEF	DEF	С	5,5
E11 (B 3103)	- G - HMX - Al	DEF	DEF	DEF	DEF	3,9
E12	- PU - HMX	DEF	DEF	DEF	DEF	3,3
E13	- PU - TATB - HMX			A	A	0,5
E14	- PU - HMX - NTO	-	-	A	С.	0,5
E15 (B 2214)	- NTO - HMX - PB	-	-	С	С	0,2

G = energetic binder PU = polyurethane binder

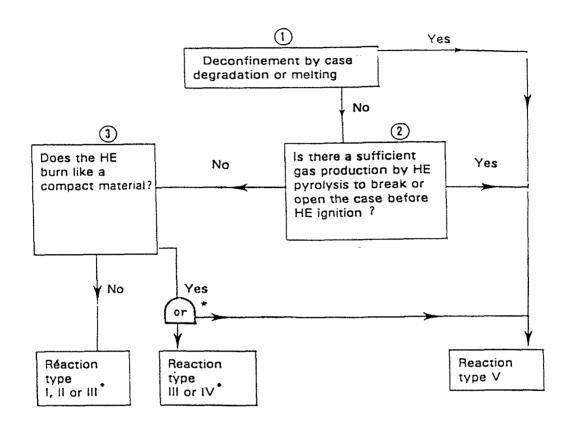
PB = HTPB binder

D = detonation
E = violent opening of the case with case fragmentation
DEF = opening of the case by caps ejection
C = combustion without opening of the case
A = absence of reaction
436

Appendix 5: Susan Test/Gap Test comparisons



Appendix 6: Hazard analysis protocol for fuel fire



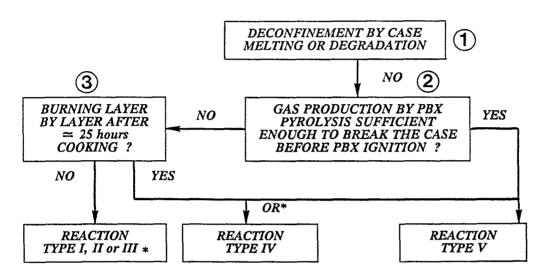
depending on confinement

Type : detonation

Type : partial detonation

II III IV V Type Type : explosion : deflagration Type : combustion

Appendix 7: Hazard analysis protocol for slow cookoff



* : Depending on confinement

Depending on confinement

Very unlikely with SCO

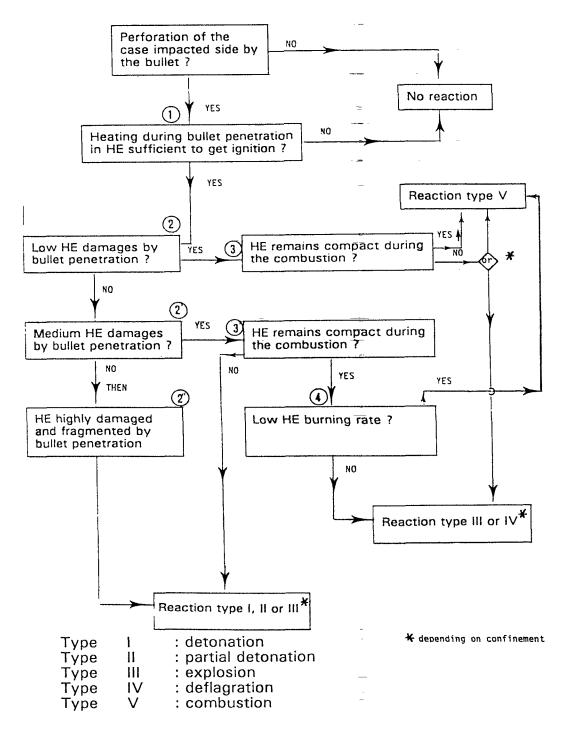
(2) and (3): According to 40 mm diameter confined pipe test

Type I : detonation

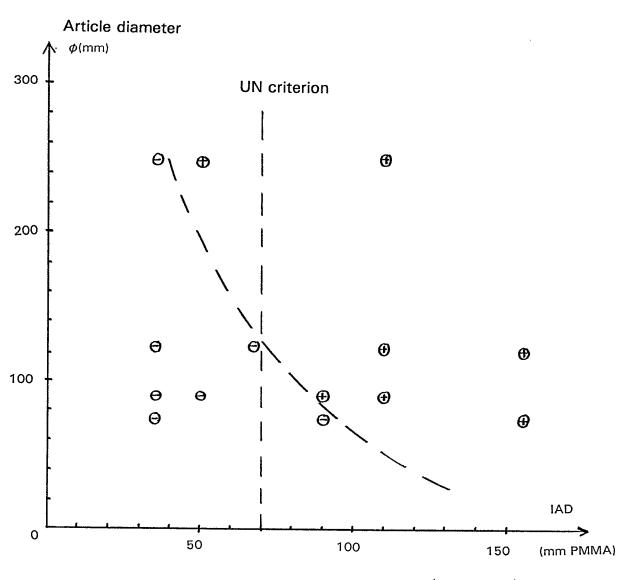
Type II : partial detonation

Type III : explosion
Type IV : deflagration
Type V : combustion

Appendix 8: Hazard analysis protocol for bullet impact on confined warhead.



Appendix 9: Comparisons between Gap Test and Sympathetic Detonation results in articles with 12.5 mm steel case.



+ : propagation

non propagation